IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

SIVIK et al.

Serial No.: 09/702,084 Examiner: P.D. Niland

Filed: October 30, 2000 Art Unit: 1714

For: COMPOSITIONS AND METHODS FOR USING ZWITTERIONIC POLYMERIC SUDS ENHANCERS

RULE 132 DECLARATION

Mail Stop AF
Commissioner for Patents
PO Box 1450
Alexandria, VA 22313-1450

Sir:

I, Jean Francois Bodet, declare as follows.

1. I have been employed by the Procter & Gamble Company since 1989. I am currently a Principal Scientist working in Dish Detergent Technology.

2. I obtained a Ph.D. degree in Physical Chemistry from University of Bordeau, France in 1985. During 1985 to 1987, I was a Post Doctorate Fellow working for Professor Theodore Davis of University of Minnesota, on Surfactant Fluid Microstructure in Microemulsions.

3. I am familiar with the above-identified application, and I am a co-inventor of the invention claimed in the above-identified application.

4. I understand the Examiner has rejected Claims 2-24 in a pending Office action. The Examiner asserts he is unclear whether the average molecular weight measured by Gel Permeation Chromatography (GPC) recited in these claims is number average, weight average, viscosity average or z average.

5. I intended that the average molecular weight of the claims to be a weight-average molecular weight. The reasons are explained in details below. A redacted copy of a Test Report of a representative sample of the presently claimed invention is attached herewith to support this statement (Exhibit A).

(i) Though it is known that various average molecular weights can be determined by GPC, the GPC instrument used by my laboratory uses a software (PD-2000) that automatically calculates the weight-average molecular weight (Mw) of the sample (See Exhibit A, Section V, Paragraph 1). Then, the software uses the calibration from an essentially monodispersed (Mw/Mn<1.10) polystyrene standard to calculate the "absolute molecular weight" of the sample (See Exhibit A, Section IV, Paragraphs 1 and 2).

(ii) On January 31, 2000, the molecular weight of a representative sample (2:1 hydroxy propyl acrylate : dimethyl amino ethylmethacrylate, designated SB99 in Exhibit A) of the presently claimed invention was determined using the above instrument and sotware (See Exhibit A). Both the Mw and Mn were calculated and reported (See Exhibit A, Page 1, second paragraph).

(iii) Moreover, the lower end of the molecular weight distribution was reported in Mw (See Exhibit A, Page 1, third paragraph "0.2% polymer with Mw < 1000 Daltons and 0.1% polymer with Mw < 500 Daltons."). This coincides with the claimed lower limit of an average molecular weight of about 1000 Daltons (See claim 2 of the present application).

The undersigned declares further that all statements made herein of his or her own knowledge are true and that all statements made on information and belief are believed to be true

and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine, or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any present issuing thereon.

Respectfully submitted,

Date: June 16, 2003 By: _____

Printed or typed name: Jean-François BODET

APV/bms
ATTORNEY DOCKET NO. APV3112

STEVENS, DAVIS, MILLER & MOSHER, L.L.P.
1615 L STREET, N.W., SUITE 850
WASHINGTON, D.C. 20036
TEL. 202-785-0100 / FAX. 202-408-5200

Attachment: Exhibit A

Molecular Weight Determination of 2:1 Hydroxy Propyl Acrylate: Dimethyl Amino Ethyl Methacrylate (SB99)

This summarizes the results of the molecular weight determination of 2:1 Hydroxy Propyl Acrylate (HPA): Dimethyl Amino Ethyl Methacrylate (DMAM) citrate polymer, i.e., SB99.

MW results of SB99 excluding contribution of citrate counterion

The weight average molecular weight (Mw) of the polymer is 29,174. The number average molecular weight (Mn) of the polymer is 7,297.

The chromatography results show that there is 0.2% polymer with Mw < 1000 Daltons and 0.1% polymer with Mw < 500 Daltons.

Analytical Conditions

0.2% solution of polymer
Mobile phase: 80/20 0.5M Ammonium Acetate/Methanol at pH 3.7
Phenomenex P5000 GFC column at 60C
Multi-Angle Laser Light Scattering detection (MALLS)

Analytical Approach

The Mw (weight average molecular weight) and Mn (number average molecular weight) of SB99 were determined using a Gel Filtration Chromatography (GFC) method on January 31, 2000 (Notebook reference MVS1174-103). The polymer was separated using a GFC column to determine molecular weight distribution. The molecular weight and distributions were measured through separation of the polymer species based on their hydrodynamic volumes.

The results obtained from the chromatography contain the contribution of the citrate counterion. There are 34% non-volatile solids in SB99-B, for which 27% is polymer and 7% is citric acid. At pH 3.7, the citrate will be coordinated with DMAM on a 1:1 basis (citric acid pKa_1 3.1, pKa_2 4.76, pKa_3 6.39)[1]. The acetate present in the mobile phase is a weaker acid than citric acid and will not be bound to DMAM.

[1] Merck Index

The Procter and Gamble Company

Supersedes: NEW	Spec. No:
Aqueous GPC Method	
Date: December 18, 1997	Issue: 1

PRELIMINARY

Determination of the Molecular Weight and Molecular Weight Distributions of PolyDMAM Polymers by Aqueous Gel Permeation Chromatography (GPC)

I. PURPOSE

This method is used to measure the "absolute" molecular weights (MW's) and MW distributions of water-soluble polyDMAM polymers.

II. OVERVIEW OF THE EXPERIMENTAL PROCEDURE

The MW and MW distributions of polymers are measured through separation of the polymer species based on their hydrodynamic volumes, which are related to their MW's.

III. APPARATUS

A. Instrumentation

HPLC System	Waters Model 600E gradient pump, combination Waters Model 410
	RI detector and a Precision Detectors PD-2000 MALLS detector
	Waters Model 717 Autoinjector,
	Precision Detectors Acquire and Analyze Software
GPC Column	Phenomenex Polysep-GFC-P5000
Column Heater	Eppendorf TC-50 or equivalent
pH Meter	Capable of meauring pH to ±0.01 unit.

Analytical Balance	Capable of weighing samples to ±0.01mg.

B. Accessories

HPLC Autosampler Vials	4 mL, glass, with Teflon caps or rubber septa
Graduated Cylinders	25mL, 1L
Volumetric Flasks	1L

C. Reagents and Chemicals

Water (DI-H_2O)	Distilled, deionized water from a Millipore, Milli-Q system or equivalent.
Ammonium Acetate	J.T. Baker or equivalent
Glacial Acetic Acid	99+% pure from J.T. Baker or equivalent
Sample Solutions	0.2% in mobile phase

D. HPLC Conditions

Mobile Phase	0.50M ammonium acetate in 1:5 Methanol:Water adjusted to pH 3.7±0.05 with glacial acetic acid. Place 1400 mL of ultra pure water in beaker. Add 38.5 g of ammonium acetate. Adjust to pH 3.7 with concentrated acetic acid. Add solution and 400 mL of methanol to a 1L volumetric flask. Complete to mark with ultra pure water.
Detection	MALLS and RI
RI Sensitivity	16x
RI Time Constant	0.2
Column Temperature	60°C
Flow Rate	0.60 mL/min

Injection Volume	200 µL
Run Time	30 minutes

IV. MALLS DETECTOR CALIBRATION

1. As discussed in Chapter 11 of the PD-2000 manual, the MALLS detector is typically calibrated using a commercial polystyrene MW standard (i.e., from Polymer Labs) that is essentially monodisperse (Mw/Mn<1.10) for which the weight average MW is known. The MW should be in the range of ~50,000 to 100,000. Page 11-3 of the PD-2000 manual lists the necessary characteristics of the calibration standard. The calibration can be done using an organic GPC column (i.e., Jordi Gel DVB Column, 10x250mm) with a THF mobile phase.

2. Through calibration of the detector, values for the 90 and 15 degree calibration constants as well as the RI constant and the inter-detector volume can be measured. These values can then be used to calculate the "absolute" MW's of other polymers in other solvents (i.e., water).

V. SAMPLE ANALYSIS

1. Using the PD-2000 acquire software, enter the concentration in mg/mL and the value of the dn/dc for the sample. If the value of dn/dc is not known, enter a value of 0.164. When the data is processed, the PD-2000 analyze software will calculate a dn/dc value for the sample that can be plugged back into the software to calculate a more representative Mw. Alternatively, the dn/dc can be experimentally determined using a differential refractometer or approximated using the tabulations given in the Polymer Handbook. The dn/dc value is to be determined.

2. After the column set has fully equilibrated with the aqueous mobile phase, inject 200µL of the sample solution onto the column set.

3. Using the PD-2000 analyze software, draw baselines for the 90 degree and RI chromatograms and select the desired integration region. Analyze the data. For samples of MW<200,000, only use the 90 degree light scattering data to calculate the "absolute" MW, since it is typically less affected by scattering due to small particles passing through the light scattering detection cell.